

# BUREAU OF ANALYSED SAMPLES LTD

Directors:-R. P. MEERES, BA (Oxon), MRSC (Managing) G. C. FLINTOFT. ACMA J. C. MEERES M. S. TAYLOR, PhD, CChem, MRSC

BRITISH CHEMICAL STANDARD CERTIFIED REFERENCE MATERIAL

# **CERTIFICATE OF ANALYSIS** BCS-CRM No. 206/3 (ECRM 453-1) **HIGH Si and P CAST IRON**

Prepared under rigorous laboratory conditions and, AFTER CERTIFICATION ANALYSIS IN GREAT BRITAIN, issued by the Bureau of Analysed Samples Ltd.

## **CO-OPERATING ANALYSTS**

## INDEPENDENT ANALYSTS

#### 1 COPPINS, W. C., MSc, FRIC,

Analyst

No. 1

2

2 SNEDDON, J.,

British Cast Iron Research Association, Scottish Laboratories, Glasgow.

Ridsdale & Co. Ltd., Middlesbrough.

ANALYSTS representing MANU

3 CLYDE, C.,

Si

3.16

3.16

st Lothian.	ton, Wes	, Livings	orks Ltd.	eron Iron W	Came	D, R. B.,		UFACTURERS and USERS BSC, Lanarkshire Works, Motherwell.						
					6.	ontent in 9	LYSES		Mean of					
	Ti	C GRAPHITIC	C TOTAL	V	Cu	As	Ni	Cr	S	Р	Mn			
-	0.040	2.39	2.41	0.051	0.107	0.019	0.070	0.055	0.051	1.62	0.73			
			2.50	0.048	0.10	0.018	0.066	0.048	0.048	1.61	0.71			
			2.45	0.048	0.106	0.022	0.068	0.050	0.049	1.64	0.73			

4 HARRISON, D. E., ARIC,

5 HICKS, C. W.,

6 MUIR, S.,

ANALYSTS representing MANUFACTURERS and USERS (cont.)

BSC, Stanton and Staveley, Nottingham.

Qualcast Ltd., Derby.

BSC, Corby Works, Corby.

$\mathbf{s}_{\mathbf{M}}$	0.03	0.02	0.02	0.003	0.004	0.003	0.002	0.01	0.003			
$\mathbf{M}_{\mathbf{M}}$	3.17	0.72	1.63	0.049	0.053	0.068	0.019	0.10	0.050			
7	3.15	0.70	1.63	0.054	0.052	0.064		0.10	0.055	2.42		
6	3.16	0.72	1.62	0.050	0.054	0.072	0.020	0.106	0.051	2.44	2.35	
5	3.21	0.72	1.63	0.047	0.054	0.067		0.098	0.051	2.47		
4	3.19	0.72	1.65	0.047	0.057	0.068	0.018	0.104	0.046	2.38		
3	3.15	0.73	1.64	0.049	0.050	0.068	0.022	0.106	0.048	2.45		

The above figures are those which each Analyst has decided upon after careful verification.

 $M_M$ : Mean of the intralaboratory means.  $s_M$ : standard deviation of the intralaboratory means.

## **CERTIFIED VALUES (Cv)**

#### mass content in %

	Si	Mn	Р	S	Cr	Ni	As	Cu	V
Cv	3.17	0.72	1.63	0.049	0.053	0.068	0.019	0.10	0.050
C(95%)	0.03	0.01	0.02	0.003	0.003	0.003	0.003	0.01	0.003

The half width confidence interval  $\mathbf{C}(95\%) = \frac{\mathbf{t} \times \mathbf{s}_{M}}{\sqrt{n}}$  where "t" is the appropriate two sided Student's t value at the 95% confidence level for "n" acceptable mean values.

For further information regarding the confidence interval for the certified value see ISO Guide 35:2006 sections 6.1 and 10.5.2.

## **DESCRIPTION OF SAMPLE**

British Chemical Standard - bottles of 100g chips graded 1700 - 250µm (10 - 60 mesh) for chemical analysis.

## BCS-CRM No. 206/3 (ECRM 453-1) HIGH Si and P CAST IRON

## NOTES ON METHODS USED

#### SILICON

Analysts Nos. 1, 3, 4, 5, 6 and 7 determined silicon gravimetrically by the British Standard Silicon Method l\*. No. 2 used a titrimetric method based on the formation of potassium fluorosilicate which is filtered off and titrated with standard alkali (Sneddon, Foundry Trade J., 1966, **120**, 605-7).

Analyst No. 2 also determined silicon gravimetrically by dehydration with perchloric acid and found 3.13%.

#### MANGANESE

Analysts Nos. 1, 3 and 4 determined manganese titrimetrically. Nos. 1 and 4 used Analoid methods, oxidising with persulphate/silver nitrate and titrating with arsenite/nitrite solution (No. 1) or arsenite solution (No. 4). No. 3 used the British Standard Manganese Method 1\* which includes a zinc oxide separation. Nos. 2, 5, 6 and 7 determined manganese photometrically after oxidation with periodate; Nos. 6 and 7 used the British Standard Manganese Method 2\*.

Analyst No. 3 also used an Analoid titrimetric method and found 0.72%.

#### PHOSPHORUS

Analysts Nos. 1, 2, 4, 5, 6 and 7 determined phosphorus titrimetrically after precipitation as phosphomolybdate. Nos. 1 and 2 precipitated according to the British Standard Phosphorus Method 1\*. Nos. 4 and 7 used the Analoid method. No. 3 used the British Standard Phosphorus Method 1\*. and completed gravimetrically as lead molybdate.

Analysts Nos. 1 and 3 also used the titrimetric Analoid method and found 1.64% and 1.65% respectively. No. 7 also determined phosphorus photometrically as phosphovanadomolybdic acid and found 1.66%.

#### SULPHUR

Analysts Nos. 1, 3 and 4 determined sulphur gravimetrically. No. 1 first carried out a chromatographic separation on an alumina column (Nydahl, Anal. Chem., 1954, **26**, 580). Nos. 3 and 4 used the British Standard Sulphur Method 1\*. The other Analysts determined sulphur by combustion. No. 2 used an air combustion method (Green, BCIRA Journ., 1968, 16, 244). No. 5 used the Strohlein method.

Analyst No. 3 also used a combustion method and found 0.050%.

#### CHROMIUM

All Analysts except Nos. 4 and 5 determined chromium photometrically with diphenylcarbazide. Nos. 1 and 6 used the Analoid method No. 51. No. 2 carried out a preliminary extraction of iron with *iso*-butyl acetate (Scholes and Smith, Metallurgia, 1963, **67**, 153). Nos. 3 and 7 used the British Standard Chromium Method 2\*. No. 4 determined chromium titrimetrically according to the British Standard Chromium Method 1\*. No. 5 used the titrimetric Analoid method No. 37.

#### NICKEL

All Analysts determined nickel photometrically with dimethylglyoxime. No. 1 carried out a preliminary separation of the bulk of the iron by extraction into diethyl ether. Nos. 3, 4, 6 and 7 used the British Standard Nickel Method 3\*.

#### ARSENIC

All Analysts precipitated elemental arsenic by hypophosphite reduction and determined it iodimetrically. Nos. 3, 4 and 6 used the British Standard Arsenic Method 1\*.

#### COPPER

All Analysts except No. 2 determined copper photometrically. Nos. 1 and 4 used the *bis*-cyclohexanone oxalyldihydrazone method (Haywood and Sutcliffe, Analyst, 1956, **81**, 651). Nos. 3, 6 and 7 used 2-2' diquinolyl according to the British Standard Copper Method 3\*. No. 2 determined copper by atomic-absorption spectroscopy.

Analyst No. 3 also used the bis-cyclohexanone oxalyldihydrazone method and found 0.105%.

#### VANADIUM

Analysts Nos. 1 and 2 determined vanadium photometrically. No. 1 used a phosphovanadotungstate method after separation of the iron by extraction into diethyl ether. No. 2 used the hydrogen peroxide method after bleaching the titanium colour with fluoride. The other Analysts determined vanadium titrimetrically. Nos. 3, 4, 6 and 7 used the British Standard Vanadium Method 1\*. No. 5 used the Analoid method No. 34.

### TOTAL CARBON

All Analysts determined carbon by combustion. Nos. 1, 3, 5 and 6 completed gravimetrically according to the British Standard Carbon Method 1\*. No. 2 completed by nonaqueous titration (Jones et al., Analyst, 1965, 90, 623; 1966, 91, 399) and No. 7 by infrared absorption.

Analyst No. 3 also used the non-aqueous titration method and found 2.45%

#### GRAPHITIC CARBON

The graphite was separated by decomposing the sample with dilute nitric acid and filtering; the carbon in the residue was determined gravimetrically by combustion according to the British Standard Carbon Method 2\*. TITANIUM

Titanium was determined photometrically with hydrogen peroxide after separation with cupferron.

\*Methods for Sampling and Analysis of Iron, Steel and Other Ferrous Metals, B.S. Handbook No. 19, first published in 1970 by the BSI, 389 Chiswick High Road, London. W4 4AL.

#### **INTENDED USE & STABILITY**

The chip sample, BCS-CRM 206/3 (ECRM 453-1), is intended for the verification of analytical methods, such as those used by the participating laboratories, for the calibration of analytical instruments in cases where the calibration with primary substances (pure metals or stoichiometric compounds) is not possible and for establishing values for secondary reference materials.

It will remain stable provided that the bottle remains sealed and is stored in a cool, dry atmosphere. When the bottle has been opened the lid should be secured immediately after use. If the contents should become discoloured (e.g. oxidised) by atmospheric contamination they should be discarded.

This Certified Reference Material has been prepared in accordance with the recommendations specified in ISO Guides 30 to 35, available from the International Standards Organisation in Geneva.

NEWHAM HALL, NEWBY,	For BUREAU OF ANALYSED SAMPLES LTD
MIDDLESBROUGH, ENGLAND, TS8 9EA	
Email: enquiries@basrid.co.uk	R.P. MEERES
Website: www.basrid.co.uk	Managing Director
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